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Abstract

© Published under licence by IOP Publishing Ltd. Comprehensive experimental results of fully oxidized (up to YBa₂Cu₃O_{6.9-7}) melt-Textured YBaCuO materials with different microstructures are presented. These microstructures are built respectively: (1) with a high dislocations density but almost without twins (after high temperature treatment at 2 GPa) and (2) with a high twin density, but practically free from dislocations and stacking faults (after high temperature oxygenation at 10-16 MPa). It is shown that for attaining high critical current densities and fields of irreversibility ($j_c(H-c, 0 T) = 9 \cdot 10^4 \text{ A/cm}^2$, $H_{irr} = 9.7 \text{ T}$ at 77 K), a high twin density in YBa₂Cu₃O_{6.9-7} matrix of MT-YBCO is required. The density of twins in fully oxidized materials depends on the distances between Y₂BaCuO₅ inclusions, larger twin densities are related to shorter distances between inclusions. The influence of phase composition of the initial powder mixtures on the distances between Y₂BaCuO₅ inclusions have been characterized and discussed.

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Correlations between the structure and superconducting properties of MT-YBaCuO

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Abstract. Comprehensive experimental results of fully oxidized (up to $\text{YBa}_2\text{Cu}_3\text{O}_{6.9-7}$) melt-textured YBaCuO materials with different microstructures are presented. These microstructures are built respectively: (1) with a high dislocations density but almost without twins (after high temperature treatment at 2 GPa) and (2) with a high twin density, but practically free from dislocations and stacking faults (after high temperature oxygenation at 10-16 MPa). It is shown that for attaining high critical current densities and fields of irreversibility ($j_c(H\parallel c, 0\text{ T})=9\cdot 10^4\text{ A/cm}^2$, $H_{\text{irr}}=9.7\text{ T}$ at 77 K), a high twin density in $\text{YBa}_2\text{Cu}_3\text{O}_{6.9-7}$ matrix of MT-YBCO is required. The density of twins in fully oxidized materials depends on the distances between Y_2BaCuO_5 inclusions, larger twin densities are related to shorter distances between inclusions. The influence of phase composition of the initial powder mixtures on the distances between Y_2BaCuO_5 inclusions have been characterized and discussed.

1. Introduction

Melt-textured $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ or Y123 (MT-YBaCuO) can operate at 77 K in magnetic bearings, MAGLEV transport, superconducting electric motors, fault current limiters, fly-wheel energy storage devices, contactless mixers for medical and biological purposes etc. To warrant these applications, the critical current density and vortex pinning should be further enhanced.

The coherence length of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ is in the range of 0.6-3.1 nm [1]. Such short coherence length leads to easily reduced transparency for the supercurrent flow through various grain boundaries [2, 3], including twinning planes. The grain boundary with large angles of misorientation poses significant obstacles to the superconducting current flow. Their presence leads to low critical current density, j_c . To avoid high-angle boundaries the material is textured using seed crystal. In parallel with the texturing (for the further increase of critical current density) small inclusions of Y_2BaCuO_5 (or



Y211) phase are formed in the almost single-domain Y123 matrix [4-8]. After texturing the Y123 matrix has deficit of oxygen and its composition is about $\text{YBa}_2\text{Cu}_3\text{O}_{6.3-6.4}$ (the material is nonsuperconducting). For the further increase of the oxygen content in Y123 matrix in order to make it superconducting, the oxygenation process can be performed in oxygen atmosphere under the ambient (0.1 MPa) or elevated (10-16 MPa) pressures [1, 9, 10].

When oxygen introduced into the basal planes of Y123 lattice the c -parameter is decreased [11] and cracking results from the associated stress. The micro-cracks parallel to ab -planes are usually formed. The difference in the expansion coefficients of Y123 and Y211 phases leads to cracks formation as well [12]. Macro-cracks are seeing after mechanical polishing of the sample surface, while micro-cracks after etching process. Besides, during oxygenation process different defects (dislocations, stacking faults, twin planes) around Y211 inclusions are formed [13].

Structural defects in superconductors are considered as pinning centers. Stacking faults and Y_2BaCuO_5 (Y211) inclusions according to [1, 14] are likely to be the more efficient in MT-YBaCuO as compared to twin planes. Dislocations are considered favorable defects for pinning as soon as there density is very high [1, 2, 3, 14, 15], and in [13] dislocations are asserted as the most important defect for pinning in MT-YBaCuO. According to [16], the smaller Y211 inclusions are, the higher numbers of dislocations are nucleated around them, the advantage of small Y211 grains in the structure of Y123. For the refinement of the Y211 inclusions some amount of platinum or cerium oxide is usually added to the initial powdered mixtures while the mechanisms of their positive effect are not clear yet.

Using the phenomenological deformation criterion of superconductivity, it have been confirmed [17] that the energy of twin boundaries and the size of the twin colonies are important factors for constructing twin morphology, which is necessary requirement for strong pinning and high critical currents. They compared the pinning forces of different types of twin structures. The ability of a crystalline defect to pin a vortex obviously depends on the size of the non-superconducting region surrounding the defect [2, 3]. Since pinning at twin boundaries in YBaCuO is highly anisotropic, it is considered that anisotropy and average pinning force make pinning at twin boundaries, unlikely to be important. After comparison of pinning abilities of a twin boundary, an intersection of twin boundary with a twin dislocation, and an intersection of twin planes, it was concluded [17, 18] that for attaining high critical currents in high magnetic fields, it is necessary to decrease the energy of twin boundaries, decrease the sizes of twin colonies and increase the area of intersections of twins.

In this work, we present the results of microstructural, superconducting and mechanical properties of two types of melt-textured YBaCuO materials (with *in-situ* formed Y211 inclusions and with additions of *ex-situ* formed Y211) oxygenated at ambient pressure (0.1 MPa, 440 °C), elevated pressure (16 MPa and 800 °C) and of high pressure-high temperature treated (at 2 GPa, 800 °C) previously oxygenated MT-YBCO, which allow us to create MT-YBaCuO structures with different concentrations of twins, stacking faults, dislocations, micro- and macro-cracks.

2. Experimental

In the present study two differently prepared MT-YBaCuO materials were used. Type 1 MT-YBaCuO was produced from precompacted $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (manufactured by Solvay), Y_2O_3 and CeO_2 , powders which were taken in the ratio $\text{Y}_{1.5}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta} + 1\% \text{ CeO}_2$ and then textured in air using $\text{SmBa}_2\text{Cu}_3\text{O}_x$ seed crystal in quasi-isothermal conditions (provided for each $40 \times 40 \times 17$ mm sample of the batch from 16 pieces) [4]. In the temperature range where the Y123 texture growth occurred, the temperature was lowered at a rate of 0.2-1.0 K/h and then cooled at 50 K/h. Type 2 MT-YBaCuO (16-30 mm in diameter 10-15 mm in height) was produced from precompacted 70 wt. % $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$, 30 wt. % Y_2BaCuO_5 and 0.15 wt. % PtO_2 powdered mixtures according to the technology described in [7, 8]. The additions of 1% CeO_2 or 0.15 wt. % PtO_2 were added to obtain finer grains of Y_2BaCuO_5 in $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ matrix during melt-texturing. After melt-texturing the sample of Type 2 had practically no macro-cracks. The absence of macro-cracks in its structure is relevant to the special technological approach: after finishing of texturing process in air the material starting from about 940 °C was cooled down in nitrogen atmosphere to prevent the oxygenation during cooling and avoid cracks formation.

The MT-YBaCuO ceramic blocks without holes are named ‘bulk’ and that with set of holes (formed by drilled or pressing perpendicular to the *ab*-planes of Y123 domain) are named ‘thin-walled’.

The principal difference between Types 1 and 2 materials is the following. In Type 1 MT-YBaCuO, the Y₂BaCuO₅ (Y211) inclusions were formed *in-situ* while in Type 2 the inclusions were formed using previously synthesized Y₂BaCuO₅ powder *ex-situ*. The difference in the materials structures can be seen in figure 1. The structure of Type 1 material contains in general finer Y211 inclusions, but they are inhomogeneously distributed. In some areas rather coarse Y211 inclusions can be found (figure 1a). In Type 2 material the Y211 inclusions are coarser, but distributed more homogeneous. There is no big difference between sizes of these inclusions. It should be mentioned that the distances between the Y211 inclusions in Y123 of Type 2 material are somewhat smaller than that in Type 1.

The oxygenation has been performed in oxygen flow at 0.1 MPa at 440 K for 270 h and at 16 MPa oxygen pressure and at 800 °C for 72 h. The heating process in the case of oxygenation at 16 MPa was started in nitrogen atmosphere at 0.1 MPa pressure, which was gradually replaced by oxygen upon rising temperature (to prevent oxygenation at low temperature and cracks formation). After reaching the highest temperature the oxygen pressure was gradually increased up to 16 MPa [9]. The YBa₂Cu₃O_{7-δ} matrices of all the materials were oxygenated up to 7-δ=6.9-7 as confirmed by the X-ray structural studies of the lattice parameters.

The high (quasi-hydrostatic mechanical) pressure-high temperature treatment [19, 20] of previously oxygenated (at 0.1 MPa and 440 °C) MT-YBaCuO of Type 1 material was performed in contact with precompacted hexagonal ZrO₂ powder at 2 GPa, 800 °C for 0.5 h. Heating in contact with ZrO₂ under 2 GPa pressure allows us to preserve oxygen content in Y123 matrix. The images of structures of high-pressure treated materials obtained under transmission electron microscopy (TEM) are shown in figure 2. The high-pressure treatment allowed increasing dislocation density up to 4 orders (from 10⁸ to 10¹² cm⁻² [21]). In the same time, the density of twins are essentially reduced, so that large volumes of the sample appear to have no twins at all, while the material remained optimally oxidized (from the point of view of the highest possible SC transition temperature for Y123 structure).

The phase composition and the crystallographic structures (amount of oxygen in Y123) were studied at room temperature by X-ray powder diffraction using a Philips X’pert” and a DRON-3 diffractometer. The X-ray patterns were taken in an angular range of 2Θ = 20–70° at a rate of 5 deg/min. The samples structures were studied using polarized light and transmission electron microscopes (figures 1-4). The Vickers microhardness and fracture toughness was estimated by indentation on *ab*-plane and in perpendicular direction (Table 1).

After texturing but before oxygenation, small rectangular bars with dimensions of about 2×2×5-7 mm were cut from both types of blocks with such orientation that longer sides of the bars were parallel to the *c*-axis of Y123 matrix and perpendicular to its *ab*-planes. It was possible because the position and orientation of seed crystal, which was used for manufacturing of melt-textured quasi-single-domain block was known. All samples were oxygenated in the same conditions simultaneously. The small rectangular bars (without cut them) were used for all characterizations presented in this work except for TEM. To estimate the critical current density, *j_c*, after oxygenation magnetization curves were obtained using small rectangular samples in VSM (vibrating sample magnetometer) for two orientations with magnetic field parallel and perpendicular to *ab*-planes. The trapped fields were studied by scanning of surfaces of the original large blocks oxygenated together with small rectangular bars using Hall probe after field cooling [10].

The amount of cracks was calculated for the entire samples using polarized microscopy. The linear densities of cracks were calculated using 10-15 separate images collected together, which demonstrated amount of cracks on the entire surfaces of the rectangular bars. After polishing macro-cracks became visible and after etching (by acid) the micro-cracks became visible as well. The amount of macro-cracks crossing 1 mm and amount of micro-cracks crossing 1 micron lengths were estimated. Usually cracks were parallel to *ab*-planes (because of *c*-parameter reduction during oxygenation) and thus they were perpendicular to the long sides of the rectangular bars. The twins

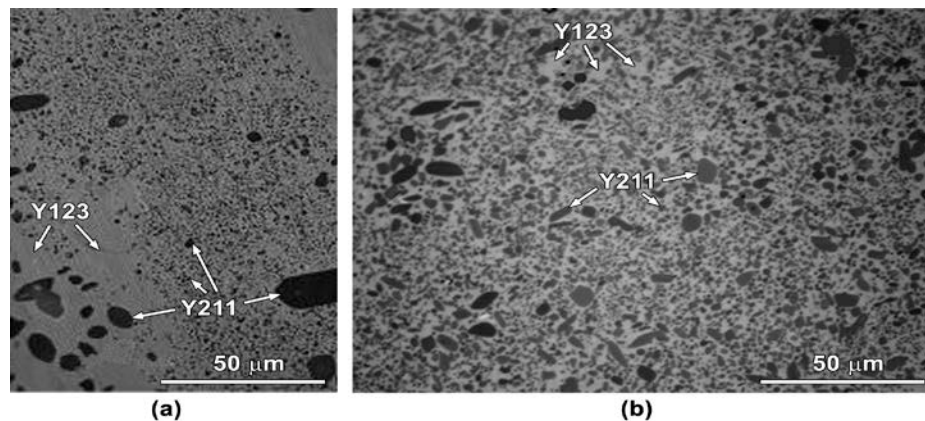


Figure 1. Structure of MT-YBCO before oxygenation: (a) – Type 1, prepared from precursor powders mixture $\text{Y}_{1.5}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ + 1 wt. % CeO_2 ; (b) – Type 2, prepared from precursor powders mixture 70 wt. % $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$, +30 wt. % Y_2BaCuO_5 + 0.15 wt. % PtO_2 .

were observed perpendicular to *ab*-plane surfaces and to micro-cracks (figures 4a, b). On *ab*-planes the colonies of twins appeared exactly perpendicular to each other (figure 3d). To minimize the effect of the materials inhomogeneity the same small sample of each material was used to estimate critical current density, amount of cracks, hardness, fracture toughness, *c*-parameter and amount of oxygen in superconducting phase, twin density in polarized light and then was cut to prepare probes for TEM.

3. Results and discussions

Table 1 summarises characteristics of the MT-YBaCuO (with $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$, $7-\delta=6.9-7$) oxygenated at different conditions and after being exposed to high-pressure treatment. Figure 2 shows the distribution of twins in MT-YBaCuO of Type 1 after high pressure-high temperature treatment and figure 3 shows the distribution of twins in Types 1 and 2 oxygenated by different ways. Figure 4 demonstrates the difference in the density of micro-cracks in Type 1 sample oxygenated under different conditions. Figure 5 shows the dependences of critical current density and pinning forces vs. magnetic fields of the materials under the study, noting possible issues in pinning force determinations [22, 23].

Analyzing the data given in figures 2-4 and Table 1, the density of twins correlates with distances between Y211 inclusions and critical current densities. As it was mentioned above, according to X-ray structural analysis the $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ matrices of all the materials investigated in this paper were fully oxygenated with $7-\delta=6.9-7$. Despite the density of dislocations after high pressure-high temperature treatment was increased by about 4 orders of magnitude and reached 10^{12} cm^{-2} and high amount of stacking faults were present, the parallel detwinning leads to low critical currents (figure 5g). The microstructure shown in figure 2b was obtained with $g=020$ (after treatment at 800°C , $P=2 \text{ GPa}$). Two types of dislocations are observed. Partial dislocations parallel to $[010]$ originated from the 211 particles and bounded stacking faults (are out of contrast). These dislocations are elongated along the screw direction. Perfect dislocations are also observed. Their main direction is parallel to $[010]$ direction but they present some steps parallel to $[110]$ and $[1-10]$ directions. Some of these steps correspond exactly to the twin domain and some are not associated to the presence of a twin boundary but bear witness of a memory effect after the detwinning process [24]. Thus, MT-YBaCuO material with high concentration of dislocations in $\text{YBa}_2\text{Cu}_3\text{O}_7$ phase but with very low twin density demonstrates critical current density essentially lower than that without dislocations and stacking faults, but with high concentration of twins (figures 3a-c, 5e, f and Table 1).

The shorter distances between Y211 grains (even in the same material, comparing figures 3a and 3b) result in a higher density of twins. The comparison of density of twins in Type 1 (inhomogeneous

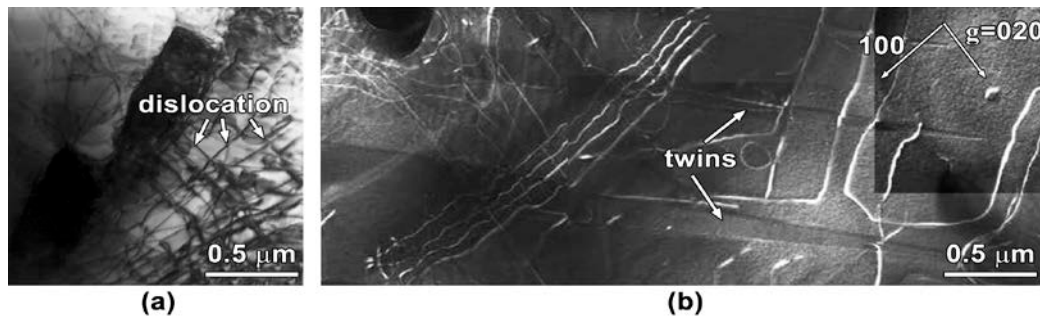


Figure 2. Structure (obtained by TEM) of Type 1 MT-YBCO after oxygenation at 0.1 MPa, and 440 °C followed by high pressure-high temperature treatment at 2 GPa, 800 °C for 0.5 h: (a) high dislocation density, (b) low twin density (detwinning).

Table 1. Characteristics of MT-YBaCuO of Types 1 and 2 after oxygenation at different conditions (in all the cases the materials $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ matrices were fully oxygenated: $7-\delta=6.9-7$)

Characteristics and conditions of oxygenation	Type 1		Type 2		Type 1 detwinned
	0.1 MPa, 440 °C	16 MPa, 800 °C	0.1 MPa, 440 °C	16 MPa, 800 °C	0.1 MPa, 440 °C, 2 GPa, 800 °C
Density of microcracks, mm^{-1}	890	200	1500	270	-
Density of twins, μm^{-1}	0.5-15	7-20	12-16	20-35	0-1
Density of macrocracks, mm^{-1}	1.5	0.4	1.3	absent	-
Critical current density at 0 T field, kA/cm^2					
$H\parallel c$	58.0	83.0	60.0	81.0	9.2
$H\parallel ab$	16.5	30.0	17.5	34.0	4.2
Field of irreversibility, T, $H\parallel c$	6.3	5.8	8.7	9.7	5.7
Maximal pinning force, N/cm^3					
$H\parallel c$	574.34	697.42	473.34	119.54	23.47
$H\parallel ab$	262.27	301.94	108.78	242.65	47.15
Magnetic field which corresponds to maximum pinning force, T					
$H\parallel c$	2.12	1.82	2.76	2.6	2.2
$H\parallel ab$	8.56	7.74	9.02	>10	8.6
Microhardness, H_v , GPa, $P=4.9$ N					
on ab	4.3±1.1	6.3±0.5	6.8±0.9	7.3±0.2	-
$\perp ab$	6.6±0.5	7.5±0.6	7.6±0.1	7.6±0.3	-
Fracture toughness, K_{Ic} , $\text{MPa}\cdot\text{m}^{0.5}$, $P=4.9$ N					
on ab	0.7±0.2	3.31±1.05	1.9±1.4	4.37±0.77	-
$\perp ab$	-	1.95	1.73±0.13	2.8±0.24	-

distribution of Y211) and Type 2 (more homogeneous distribution of Y211 and somewhat shorter distances between the inclusions) prove the statement as well. The increase in twin density correlates with the increase of pinning force maximum (Table 1). The figure 3d shows the twins in Type 1 material in polarized light. The magnification of optical microscope was not enough to resolve twins between small Y211 inclusions located from each other on short distances in Type 1 material. We did not succeed to see clearly twins in Type 2 materials (with high density of Y211 inclusions and short distances between them) using polarized light microscope, but TEM observations showed that the twin density was very high ($20-35 \mu\text{m}^{-1}$) and that dislocations were practically absent (5 probes from the

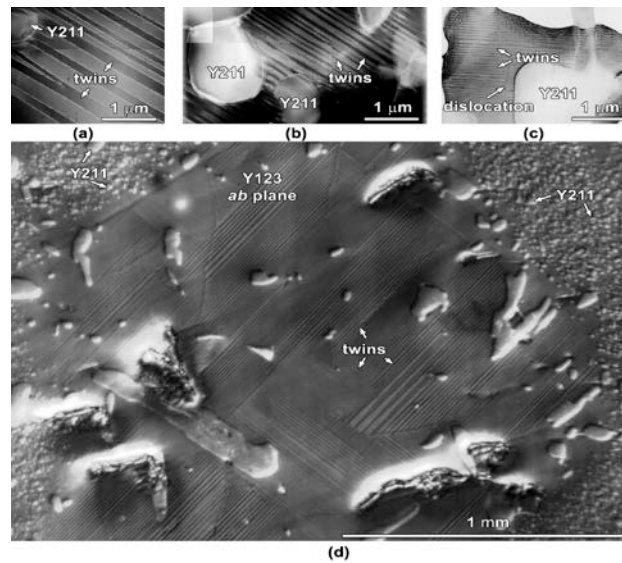


Figure 3. Twins in MT-YBaCuO structures: (a, b, c) - images obtained by TEM after oxygenation at 16 MPa, 800 °C for 72 h (a) and (b) - of Type 1 material, (c) - of Type 2 material; (d) – MT-YBaCuO of Type 1 after oxygenation at 0.1 MPa, at 440 °C for 270 h (image obtained using optical polarized light microscope).

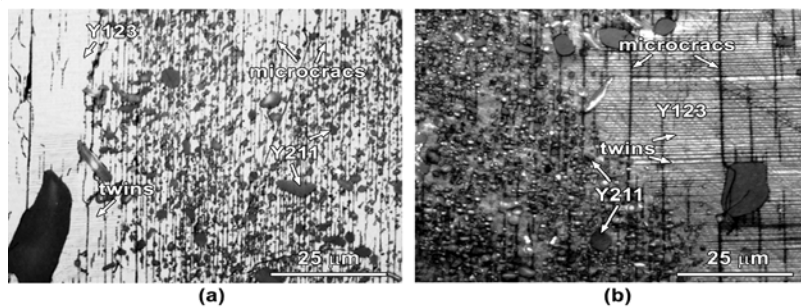


Figure 4. Micro-cracks in MT-YBaCuO of Type 1 oxygenated at 0.1 MPa, at 440 °C for 270 h (a) and at 16 MPa, 800 °C for 72 h (b).

same sample were studied by TEM). It seems that pinning occurred in the places where twin walls meet Y211 inclusions.

The figure 4 demonstrates difference in micro-cracking of Type 1 material oxygenated under high and low temperatures. The essential reduction of density of micro-cracks has been observed for Type 2 material as well (when oxygenation was performed under high temperature, see Table 1). The reduction in density of micro-cracks can be the reason of j_c increase in c direction ($H||ab$) in the materials oxygenated under high temperatures what leads to the decrease of anisotropy of j_c and to the improvement of mechanical characteristics (microhardness and fracture toughness, Table 1).

The shorter distances between Y211 inclusions results in higher density of micro-cracks. Likely it is the reason for j_c in c direction ($H||ab$) for Type 2 material to be is somewhat lower than that for Type 1 (Figures 4a and 4b, 4e and 4f). The elevated pressure (10-16 MPa) is necessary to keep oxygen in Y123 structure at high (800 °C) temperature. Since the diffusion rate increases at high temperatures, and the oscillations of atoms around their positions in crystal lattice increase, oxygen under enhanced pressure can enter the $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ tetragonal structure with lower resistance. This leads to the disappearance of macro-cracks and a significant decrease in micro-cracks density and to the reduction of the time of oxygenation. The trapped field of MT-YBaCuO thin-walled block (16 mm in diameter, 10 mm in height with 0.8 mm holes) oxygenated at 16 MPa, 800 °C for 72 h was 0.54 T. It was 2 times higher than that of long-term oxygenated block at 0.1 MPa and 440 °C of the same sizes.

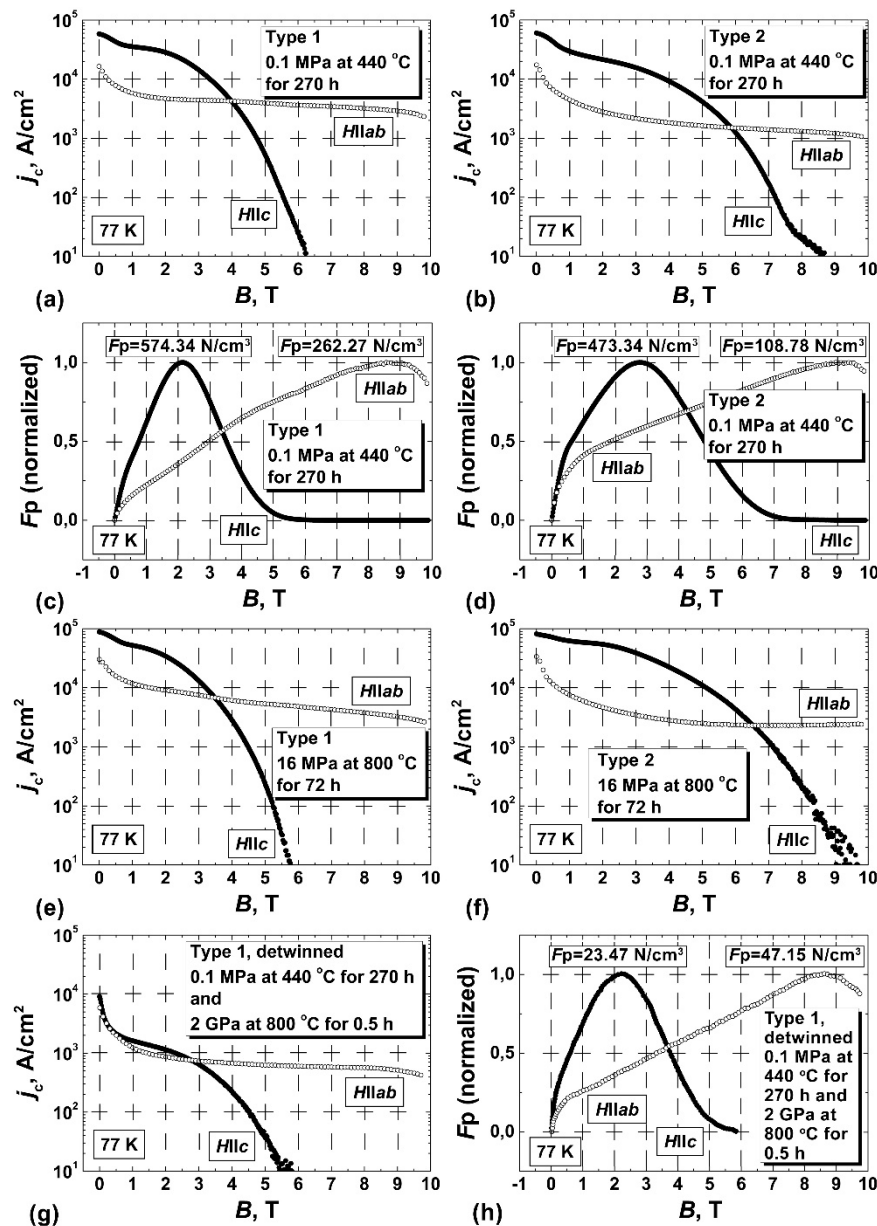


Figure 5. Dependences of critical current densities, j_c , vs magnetic field, B , for MT-YBaCuO, when magnetic field is parallel and perpendicular to ab planes of matrix $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ phase: (a), (b) – of Types 1 and 2, respectively, after oxygenation at 0.1 MPa, at 440 °C for 270 h; (e), (f) of Type 1 and 2, respectively, after oxygenation at 16 MPa, 800 °C for 72 h; (g) – of Type 1 after oxygenation at 0.1 MPa, and 440 °C and then treatment at 2 GPa, 800 °C for 0.5 h; (c, d, h) – Normalized pinning forces, F_p , vs magnetic fields, B , of MT-YBaCuO: oxygenated at 0.1 MPa, at 440 °C for 270 h of Types 1 (c) and 2 (d); and oxygenated at 0.1 MPa, 440 °C and then treated at 2 GPa, 800 °C for 0.5 h (h). The maximum absolute F_p values are written over each curve and given in Table 1.

4. Conclusions

In MT-YBaCuO twins have been experimentally proved to play a more significant contribution than dislocations and stacking faults in achieving high critical current densities. We also showed that the densities of twins and micro-cracks (parallel to the ab -planes) in the $\text{YBa}_2\text{Cu}_3\text{O}_7$ matrix correlate with the distances between Y_2BaCuO_5 inclusions, usually the density of twins and micro-cracks are higher if

distances between Y_2BaCuO_5 inclusions located in $\text{YBa}_2\text{Cu}_3\text{O}_7$ matrix are smaller. The oxygenation under high temperature (800 °C) under elevated oxygen pressure leads to the high twin density, reduction of micro-cracks, dislocations and stacking faults. The high temperature oxygenation under oxygen pressure result in an j_c increase and a decrease in its anisotropy, as well as in an increase of the materials mechanical characteristics (hardness and fracture toughness). Larger trapped fields and a reduced time of oxygenation result also from this process treatment.

Acknowledgments

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